EXTRACTION AND CHARACTERIZATION OF ESSENTIAL OIL FROM GINGER (*ZINGIBER OFFICINALE* ROSCOE) AND LEMONGRASS *(CYMBOPOGON CITRATUS)* BY MICROWAVE-ASSISTED HYDRODISTILLATION(MAHD)

RANITHA A/P MATHIALAGAN

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FACULTY OF CHEMICAL AND NATURAL RESOURCES ENGINEERING
UNIVERSITI MALAYSIA PAHANG

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ABSTRACT

Microwave-assisted hydrodistillation (MAHD), an advanced distillation method that take advantage of microwave heating with the conventional hydrodistillation, recently been widely attended for the extraction of essential oil from medicinal plants and herbs due to its economic and green technology. One way in maximizing the efficiency of a method is optimizing its parameter condition to obtain maximum yield. In that case, this study was carried out to investigate the performance of MAHD in the extraction of essential oil from Ginger (*Zingiber Officinale* Roscoe) and Lemongrass (*Cymbopogon citratus).* The effect of three main factors which were microwave power, extraction time and water to raw material ratio were investigated to optimize the extraction operating conditions for obtaining maximum oil yield. As a result the best condition that has been determined for maximum essential oil production were under 250 W microwave power for 90 minutes at water to raw material ratio of 8:1. This optimum condition was finalized based on its maximum yield from Ginger (*Zingiber Officinale* Roscoe) and Lemongrass (*Cymbopogon citratus)* which were 0.85% (w/w) and 1.37%(w/w), respectively. Then, the oil samples at different extraction time were analyzed to evaluate its quality by determined its chemical constituent through GC-MS. The main components detected in the essential oil of Ginger (*Zingiber Officinale* Roscoe) were Borneol, β-Bisabolene, Cineole, α-Cedrene, α-Curcumene, β-Farnesene (E), β-Sesquihelladiene, β-Thujene and Zingiberene. Wherelse, the main components in the essential oil of Lemongrass (*Cymbopogon citratus)* were Citral, Geranic Acid, Geranyl Acetate, Linalool, Neric acid, (Z) Citral, β-mycrene and β-Thujene. The dominant component in the essential oil of Ginger (*Zingiber Officinale* Roscoe) and Lemongrass (*Cymbopogon citratus)* were Zingiberene and Citral, respectively. The maximum oil yield from Ginger (*Zingiber Officinale* Roscoe) and Lemongrass *(Cymbopogon citratus)* were obtained at shorter extraction period without significant affect on their chemical constituents. This may prove MAHD as a cost effective method and also expected would bring volume of worth in the essential oil production industry.

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ABSTRAK

Pergabungan microwave dengan penyulingan berasaskan air (MAHD) adalah suatu teknlogi terkini yang menggunakan kelebihan gelombang mikro dalam aplikasi penyulingan hidro tradisional yang sejak kebelakangan ini digunakan secara berleluasa dalam pengekstrekan minyak pati tumbuhan herba kerana kosnya yang rendah serta teknologinya yang mesra alam. Salah satu cara untuk memaksimakan kebolehan sesuatu kaedah adalah dengan mengoptimumkan faktor-faktor yang mempengaruhi penghasilan minyak pati untuk memperoleh hasil yang tinggi.Oleh yang demikian, penyelidikan ini telah dijalankan untuk menyiasat kebolehan MAHD dalam pengekstrekan minyak pati halia dan serai. Tiga fakto utama iaitu kuasa gelombang mikro, masa ekstrasi dan nisbah air kepada bahan mentah telah dikaji untuk mengenalpasti kombinasi terbaik untuk menghasilkan jumlah minyak pati yang tinggi.Dengan itu, kombinasi pembolehubah yang memberikan hasl minyak yang terbanyak telah dikenalpasti sebagai kuasa gelombang mikro 250W, masa pengekstrekan selama 90 minit dan nisbah air kepada bahan mentah 8:1. Keadaan ini boleh digunapakai untu kedua-dua tumbuhan yang dianalisa dalam penyelidikan ini. Kombinasi kedaan pembolehubah ini telah dimuktamadkan sebagai keadaan yang teroptimum berdasarkan jumlah minyak yang dihasilkannya iaitu 0.85%(w/w) bagi halia dan 1.37% bagi serai. Kemudian, minyak pati yang dihasilkan telah dianalisa dengan menggnakan GC-MS untuk mengenalpasti komponen-komponen kimia yang terkandung dalam miyak tersebut bagi mengetahui paras kualitinya. Komponen-komponen kimia utama yang dikenalpasti dalam minyak pati halia adalah Borneol, β-Bisabolene, Cineole, α-Cedrene, α-Curcumene, β-Farnesene (E), β-Sesquihelladiene, β-Thujene dan Zingiberene. Manakala komposisi utama minyak pati serai adalah Citral, (Z) Citral, β-mycrene dan β-Thujene. Komponen yang dominan bagi halia adalah zingiberene dan bagi serai adalah citral. Hasil minyak pati yang tinggi telah tercapai dalam masa pengekstrekan yang rendah melalui kaedah MAHD ini tanpa sebarang penjejasan keatas komposisi kimianya. Ini membuktikan MAHD sebagai suatu kaedah pengekstrekan yang berkos rendah serta ia diramalkan dapat membawa banyak kebaikan dalam indutri penghasilan minyak pati.

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TABLE OF CONTENTS

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LIST OF TABLES

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LIST OF FIGURES

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LIST OF SYMBOLS

˚ C	Degree Celsius
%	Percentage
kPa	Kilo-Pascal
Hz	Hertz
GHz	Giga-Hertz
W	Watts
ml	Mili-Liter
g	Grams
L	Liter
min	Minutes
hr	Hours
m	Meter
mm	Mili-Meter
µL	Micro-Liter
cm	Centimeter
mL/min	Mili-Liter Per Minute
w/w	Weight of Oil/Weight of Plant Materials
$	Dolar
RM	Ringgit Malaysia
V	Voltage
α	Alpha
β	Beta

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LIST OF ABBREVIATIONS

HD	Hydrodistillation
MAHD	Microwave-Assisted Hydrodistillation
GC-MS	Gas Chromatography-Mass Spectrometer
MAE	Microwave-Assisted Extraction
FC	Foot Cell
SC	Stalk Cell
MHC	Mother Headcell
HC	Head Cell
GC	Gas Chromotograph
MS	Mass Spectrometer
EID	Electron Impact Deionization

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LIST OF APPENDICES

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CHAPTER 1

INTRODUCTION

1.1 BACKGROUND OF STUDY

Essential oil referred to any concentrated, hydrophobic (immiscible with water), typically lipophilic (oil or fat soluble) liquid of plants that contains highly volatile aroma compounds and carries a distinctive scent, flavor, or essence of the plant. This large and diverse class of oils also is referred to as volatile oils or ethereal oils. Essential oils are found in diverse parts of plant including leaves, seeds, flowers, roots and barks. For the plant, essential oils are thought to be vital for the life of the plant, containing compounds that help to fight parasites and infections; many essential oils have anti-bacterial, anti-fungal, and anti-parasitic properties. For people, essential oils are used in perfumes, cosmetics, and bath products, for flavoring food and drink, for scenting incense and household cleaning products, and for medicinal purposes. Interest in essential oils has revived in recent decades, with the popularity of aromatherapy, a branch of alternative medicine which claims that the specific aromas carried by essential oils have curative effects.

Zingiber Officinale Roscoe or its common name ginger is a perennial herb and grows to about 3 - 4 feet high with a thick spreading tuberous rhizome. Every year it shoots up a stalk with narrow spear-shaped leaves, as well as white or yellow flowers growing directly from the root. The name ginger is said to be derived from Sanskrit word srngaveram meaning "horn root" with reference to its appearance. The plant is said to originate from India, China and Java, yet is also native to Africa and the West Indies. They are commonly used as ornamentals, as spices, and for their medicinal properties. Gingers are distinguished by the presence of a labellum, formed by the fusion of two sterile stamens, and by the presence of essential oils in

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their tissues. The characteristic odor and flavor of ginger is caused by a mixture of zingerone, shogaols and gingerols, volatile oils that compose one to three percent of the weight of fresh ginger. Ginger oil can vary in color from pale yellow to a darker amber color and the viscosity also ranges from medium to watery. The essential oil has various chemical constituents including the following which are a-pinene, camphene, b-pinene, 1,8-cineole, linalool, borneol, y-terpineol, nerol, neral, geraniol, geranial, geranyl acetate, b-bisabolene and zingiberene. The oil is extracted by means of steam distillation from the unpeeled or dried, ground-up root (rhizome) of the plant, and can yield about 2 - 4 % oil.

Cymbopogon citratus which is also commonly known as lemongrass, a native of India, is comes from family of fragrant grasses. *Cymbopogon* is a tall, aromatic perennial grass that can grow up to 90 cm in height and 5 mm wide. Fresh lemongrass contains approximately 0.4% volatile oil and rests are non-volatile components and nutritious such as calcium, iron, magnesium, manganese, phosphorus, potassium, selenium and zinc. Lemongrass essential oil is extracted from the fresh or partly dried leaves by steam distillation. It has a lemony, sweet smell and is dark yellow to amber and reddish in color, with a watery viscosity. The main chemical components of lemongrass oil are myrcene, citronellal, geranyl acetate, nerol, geraniol, neral and traces of limonene and citral. The essential oil from Ginger (*Zingiber Officinale* Roscoe) and Lemongrass *(Cymbopogon citratus)* used for same purposes which for culinary and medicinal. Lemongrass oil also use in perfumes and as insect repellent.

The main methods to obtain essential oils from the plant materials are hydrodistillation (HD), steam distillation, steam and water distillation, maceration, empyreumatic distillation, and expression. Among these methods, HD has been the most common approach to extract the essential oils from the medicinal herbs plants. However, in order to reduce the extraction time and possibly improve the extraction yield, to enhance the quality of the approaches such as microwave-assisted extraction (MAE), pressurized solvent extraction, supercritical fluid extraction, and ultrasound-assisted extraction have also been sought. In an attempt to take advantage of microwave heating with the conventional HD, microwave-assisted hydrodistillation (MAHD) was then developed and used for the extraction of essential oils from some

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plants. Part of them are extraction of essential oils from *Satureja hortensis* and *Satureja Montana* (Rezvanpanah *et al*.,2008), Mango (*Mangifera indica* L.) flowers (Wang *et al.*,2010) and from *Thymus vulgaris* L. (Golmakani *et al.*, 2007). However, there is no research yet been carried out of extracting essential oil from Ginger (*Zingiber Officinale* Roscoe) and Lemongrass *(Cymbopogon citratus) by* MAHD. Therefore, this study will be carry out to investigate the potential of MAHD for the extraction of essential oils from Ginger (*Zingiber Officinale* Roscoe) and Lemongrass *(Cymbopogon citratus)* besides evaluating the extraction time and its cost.

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1.2 PROBLEM STATEMENT

The worldwide market for essential oils has been estimated at US$2.6 billion, with an annual growth rate of 7.5 percent. It is projected that the value of the global market for herbal products would reach US$200 billion by the year 2008 (The Sun, 2001). In Malaysia, the herbal market was estimated to be worth RM2.5 billion annually, with the local herbal industry capturing only 5 percent to 10 percent of the market. Moreover, the herbal industry is expected to be the main contributor to the country's income in the future (Berita Harian, 2001).

This increasing demand of essential oil, such as ginger oil and lemongrass oil has opened up wide opportunities for global marketing and this leads to the requirement of competitive product in market which comes with all the advantages in term of cost, quality and its production time. As stated before essential oil is a volatile component. Therefore, it is vital to identify a best extraction technique, so that a higher quality essential oil with higher yield can be extracted.

Hydrodistillation (HD) is the most common approach to the extraction of essential oils from medicinal herbs and plants. However, these conventional methods present several drawbacks such as long extraction times, potential loss of volatile constituents, high energy use, and so on. Thus, it is not suitable with the current market requirement. Therefore, developing an alternative extraction technique that is rapid, sensitive, safe, and energy-efficient is highly desirable. As the result, to improve this existing extraction process, a more active and efficient enhancement can be added and microwave is one of them.

Microwave-assisted hydrodistillation (MAHD) method is a more recent technique used to recover volatile components such as essential oil. In this method, plant material placed in a Clevenger apparatus is heated inside a microwave oven for a short period of time to extract the essential oil where heat is produced by microwave energy. The sample reaches its boiling point very rapidly, leading to a very short extraction or distillation time. With the microwave distillation technique it is possible to achieve distillation with the indigenous water of the fresh plant material (Kürkçüoğlu, 2010).

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Although the effect of microwave-assisted hydrodistillation has been conduct on various essential oil extraction, its effect on Ginger (*Zingiber Officinale* Roscoe) and Lemongrass *(Cymbopogon citratus)* , has not yet been explore yet and therefore this research will conducted. Further priority is given on the factors that can influence the extraction time and operation cost.

1.3 OBJECTIVES

The main objective of the present research are to identify the chemical composition of the essential oil from Ginger (*Zingiber Officinale* Roscoe) and Lemongrass *(Cymbopogon citratus)* which were extracted by using microwae-assisted hydrodistillation method (MAHD). A second objective was to investigate the performance of MAHD on the yield of essential oil. Finally, the objective of this research is to identify the effect of extraction time and operation cost.

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1.4 SCOPE OF STUDY

1.4.1 To analyze the chemical composition of essential oil by using Gas Chromotography- Mass Spectrometry (GC-MS).

1.4.2 To study the performance of microwave-assisted hydrodistilation method in extraction of essential oil from Ginger (*Zingiber Officinale* Roscoe) and Lemongrass *(Cymbopogon citratus).*

1.4.3 To study the effect of extraction time to the yield of essential oil from Ginger (*Zingiber Officinale* Roscoe) and Lemongrass *(Cymbopogon citratus).*

1.4.4 To find the extraction yield and efficiency of MAHD method.

1.4.5 To study the operational cost based on extraction time and yield.

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1.5 RATIONALE AND SIGNIFICANCE

The rationale of this proposed research project is to examine the performance of Microwave-Assisted Hydrodistillation method in the extraction of essential oil from Ginger (*Zingiber Officinale* Roscoe) and Lemongrass *(Cymbopogon citratus)* based on its yield and efficiency. The results of this research would signify the identification of a best extraction method for the production of essential oil from Ginger (*Zingiber Officinale* Roscoe) and Lemongrass *(Cymbopogon citratus)* which demand is increasing from year to year in global market for numerous applications.

There are various advantages of using Microwave-Assisted Hydrodistillation method for this extraction purpose. Although the distillation was accomplished in a shorter time, an oil yield through this process is slightly high compared to the conventional extraction method and this would go well to supply the ever increasing rate of demand for essential oil from ginger and lemongrass. This shorter period consumption for extraction leads to lower power consumption and this reduce the operating cost as well. In addition, MAHD also doesn't utilize any chemicals. Therefore, the essential oil extracted from this method is essentially pure and safe. These criteria are very important for essential oil such as ginger oil and lemongrass oil since they are highly employed for culinary and medicinal purposes.

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CHAPTER 2

LITERATURE REVIEW

2.1 ESSENTIAL OIL

Essential oils were mankind's first medicine. They have a long history, being used by the ancient civilizations of Egypt, Greece, India, and Rome; more than 5,000 years ago, the ancient civilizations of Mesopotamia utilized machines for obtaining essential oils from plants. Today modern science is rediscovering the wisdom of the ancients. Essential oils are able to reach deep into the recesses of our brains, cross over the chemical barriers, and open the hidden channels within our minds and bodies. Essential oils fragrances pass on to the limbic system of the brain without being registered by the cerebral cortex. Within the limbic system resides the regulatory mechanism of the innermost core of our being. Since the limbic system is directly connected to those parts of the brain that control heart rate, blood pressure, breathing, memory, stress levels, and hormone balance, essential oils can have some very profound physiological and psychological effects. An essential oil is a liquid that is generally steam or hydro-distilled from flowers, leaves, bark and roots of plants and trees and are the compounds responsible for the aroma and flavor associated with herbs, spices, and perfumes.

Essential oils may have two major components which are terpene hydrocarbon, and oxygenated compounds terpene hydrocarbon can be divided into two group; monoterpenes and sesquiterpenes. While oxygenated compounds are phenols, monoterpenes, and sesquiterpenes alcohols, aldehydes, ketons, esters, lactones, coumarins, ethers, and oxides. Monoterpenes compounds are found in nearly all essential oil and have a structure of 10 carbons atoms and at least one

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double bond. The 10 carbons atoms are derived from two atoms isoprene units. Monoterpenes react readily to air and heat sources. These components have anti-inflammatory, antiseptic, antiviral, and antibacterial therapeutic properties. Sesquiterpenes consist of 15 carbons atoms and have complex pharmacological actions. It has anti-inflammatory and anti-allergy properties. There are three main aromatic groups which are phenols, terpenes alcohols, and aromatic aldehydes.

The essential oils in aromatic herbs are known to be largely located within the glandular structures that develop on the surface of leaves and other organs of the plants. The peltate hairs appear to contain most of the oil and are henceforth called 'the glands'. Each gland originates from a single protodermal cell that undergoes division and derives two unequally sized cells. The lower cell corresponds to the foot cell (FC) while the upper daughter cells re-divides to yield the stalk cell (SC) and the mother headcell (MHC). The foot and stalk cell remain unicellular throughout the subsequent development of the gland while the mother cell of the head further divides to give rise to 8 or 12 head cells (HC) by the end of the development. Climatic factors, rates of plant metabolism, differentiation and secretory activity of glandular hairs affect synthesis and secretion of essential oils. Following figure illustrate the developments of these three glands.

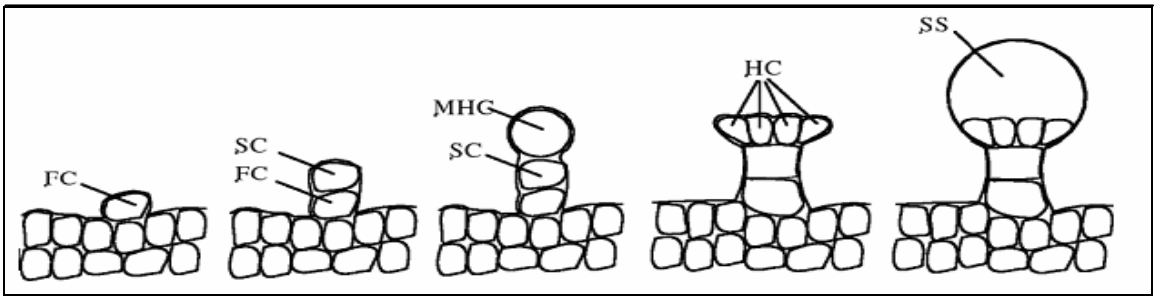

Figure 2.1: Developments of glands

Source: James 2005

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2.2 AN INTRODUCTION OF GINGER (*Zingiber Officinale* Roscoe)

The botanical name of ginger plants, as mentioned earlier, is *Zingiber officinale*. It is thought to come from the Sanskrit word *singabera* which was from Arabic and Greek words meaning 'shaped like a horn'. It probably got its name because the rhizomes look like deer's antlers. It is also known by various names such as African ginger, black ginger, sunthi, East Indian pepper, Jamica pepper, German ingwer, Italian zenzero jengibre, myoga, zangvil, gingembre, dinnsear, engifer, shouga, imbir, luya and gung.

Table 2.1: Taxonomy of Ginger (*Zingiber Officinale* Roscoe)

Kingdom	*Plantae*
Subkingdom	*Viridaeplantae*
Phylum	*Tracheophyta a*
Subphylum	*Spermatophytina*
Intraphylum	*Angiosperma*
Division	*Magnoliphyt*
Class	*Liliopsida*
Order	*Zingiberales*
Family	*Zingiberaceceae*
Genus	*Zingiber*
Species	*Officinale*
Scientific name	*Zingiber officinale*
Common name	Ginger

Source: Farlex (2004)

An essential oil is a liquid that is generally steam or hydro-distilled from flowers, leaves, bark and roots of plants and trees and are the compounds responsible for the aroma and flavor associated with herbs, spices, and perfumes. Essential oils molecules are made up primarily of carbon, hydrogen, and oxygen. The aromatic constituents of essential oils are built from hydrocarbon chains. The basic building

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block of many essential oils is a five-carbon molecule called an isoprene which built most of the essential oils.

The part of the Ginger (*Zingiber Officinale* Roscoe) plant commonly known and consumed is the underground stem, or rhizome, although it is often referred to as "ginger root". This part of the plant stores its food reserves, and is the one used for both cooking and medicinal purposes. The stem grows up to about 12 inches above the ground and has long, ribbed, green leaves, with yellow or white flowers. Ginger flowers have also been described as being greenish yellow and streaked with purple down the sides.

The strong taste and stimulating effects of Ginger (*Zingiber Officinale* Roscoe) on the body are largely down to the presence of an oily substance called gingerol as well as volatile oils. Gingerols and shogaols present in Ginger (*Zingiber Officinale* Roscoe) as pungent chemical substances. Ginger (*Zingiber Officinale* Roscoe) also contains some amount of essential oils in the root, which is the reason for its fragrance.

To grow Ginger (*Zingiber Officinale* Roscoe), the rhizome is simply planted in the ground and a new plant springs up. Ginger can actually grow in many places, but moist regions near the equator are considered best. As ginger ages, the amount of essential oils increases. So, the intended use of the rhizome determines when it is harvested. If it is for use as fresh or preserved ginger, it might be harvested when it is about 5 months old where at this time the plants have not yet matured. The rhizomes are still tender and not quite as pungent. Dried ginger calls for a more pungent aroma so those plants might be harvested at 8 or 9 months. If it is the essential oils that one is after, the plant might be harvested even after 9 months. Ginger is traditionally harvested by hand although there are mechanical diggers made just for this purpose. China is said to be largest producer of ginger today, followed by India.

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Figure 2.2: Schematic Diagram of Ginger (*Zingiber Officinale* Roscoe)

Source: Paul Gates 2008

2.2.1 History of Ginger (*Zingiber Officinale* Roscoe)

The scientific name of Ginger which is *Zingiber officinale* was given by the English botanist, William Roscoe (1753-1831) in an 1807 publication. The history of Ginger goes back over 5000 years when the Indians and ancient Chinese applied it as a tonic root for all ailments. This proved when referred back in the Hindu epic Mahabharata written around the 4th century BC describes a meal where meat is stewed with ginger and other spices. It was also an important plant in the traditional Indian system of Ayurvedic medicine.

Besides that, since 2000 years ago ginger was also highly important as an article of trade and was exported from India to the Roman Empire where it was valued more for its medicinal properties than as an ingredient in cookery. It continued as an article of trade to Europe even after the fall of the Roman Empire, with Arab merchants controlling the trade in ginger and other spices for centuries.

Along with black pepper, ginger was one of the most commonly traded spices during the 13th and 14th centuries. Arabs carried the rhizomes on their voyages to East Africa to plant at coastal settlements and on Zanzibar. During this time in England, ginger was sought after, and one pound in weight of ginger was equivalent to the cost of a sheep.

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